TECHNOLOGY DEVELOPMENTS TOWARDS A COMBINED XRD-XRF INSTRUMENT FOR PLANETARY SURFACE ANALYSIS

Dr Nick Nelms

Space Research Centre

University of Leicester

UK

4th International Planetary Probe Workshop June 27-30, 2006, Pasadena

N. Nelms[1], A. Holland[2], D. Talboys[1], I. Hutchinson[2], D. Simpson[2], G. Cressey[3], P. Bland[4]

[1]Space Research Centre, University of Leicester, Leicester, LE1 7RH, UK.

[2]e2v centre for electronic imaging, Brunel University, Uxbridge, UB8 3PH, UK.

[3]Dept. Mineralogy, The Natural History Museum, Cromwell Road, London, SW7 5BD, UK.

[4]Dept. Earth Science & Engineering, Royal School of Mines, Imperial College, London, SW7 2AZ, UK.

X-ray Diffraction

Why use an X-ray Diffractometer?

In the case of Mars

- No diffractometer has flown to date – base-lined for both MSL (Chemin) and ExoMars (MarsXRD)
- Previous geochemical measurements made using X-ray fluorescence (XRF) or alpha particle back-scattering (e.g. Viking, Sojourner and MER)
- This provides elemental composition only from which mineralogy must be inferred – different minerals can have the same elemental composition
- XRD measures the mineralogy directly by looking at coherent scatter from the crystal lattice components
- Used efficiently an XRD instrument can also simultaneously measure XRF as well

X-ray Diffraction

Why determine the mineralogy?

- Mineralogy constrains process - a geologist needs mineralogy as well as chemistry in order to understand a planet's history

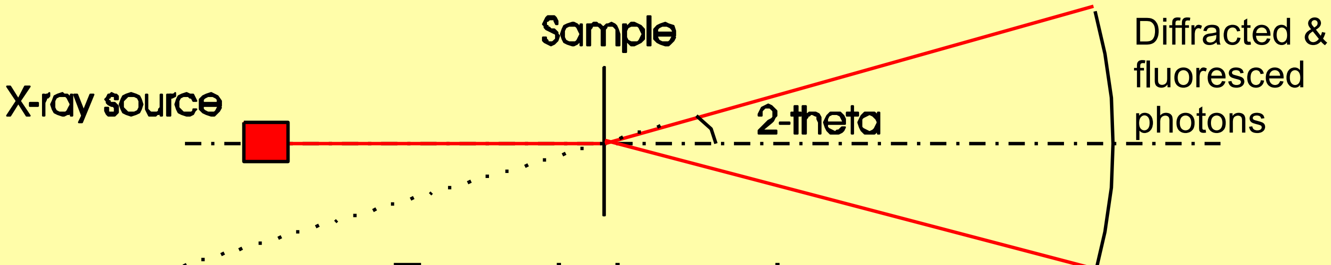
Example: Mars -The Water Story

Combined XRD-XRF is capable of

- Determining precise mineralogy of aqueous minerals
- Examining the riddle of the missing carbonates – seen in Martian meteorites but not found on the surface
- Exposing the structural nature of quartz – important geologically – can be aqueously deposited - XRF alone can’t help

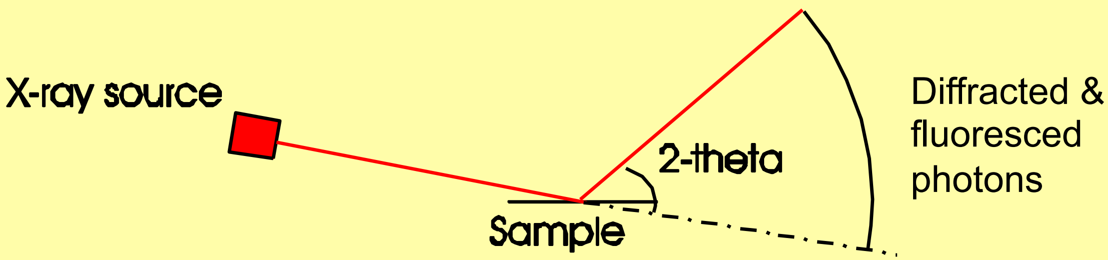
X-ray Diffraction

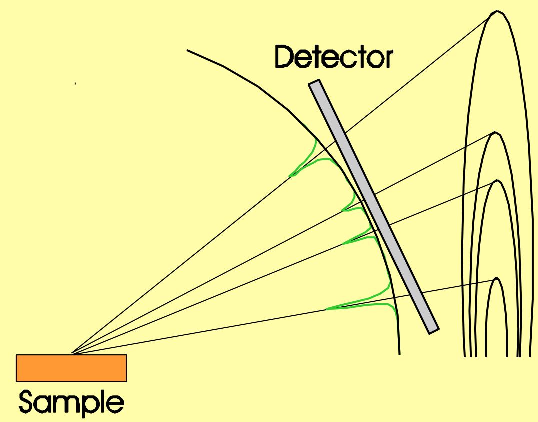
What does XRD geometry look like?



Transmission mode

Reflection mode

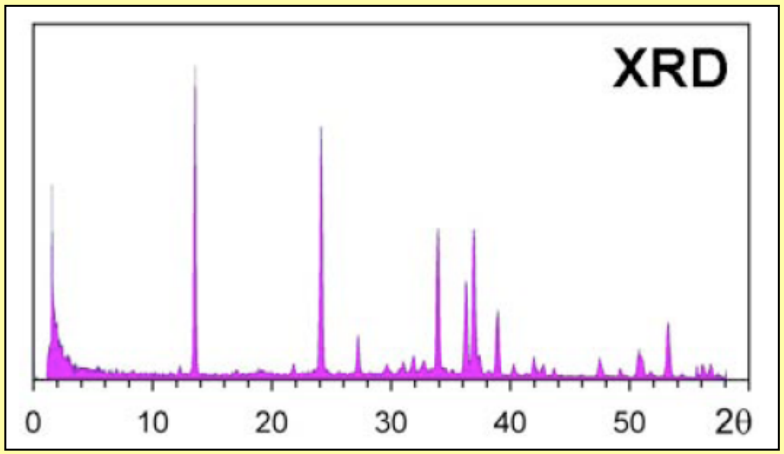




X-ray Diffraction

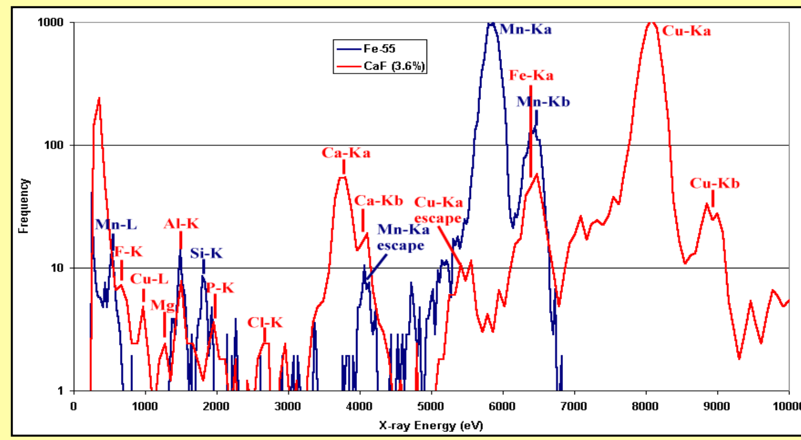
XRD – XRF output

Sample XRD plot



2θ angle

Sample XRF plot



Energy

X-ray source

Requirements

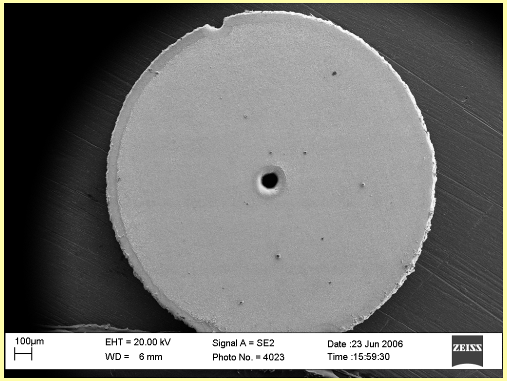
Coherent X-ray source that is monochromatic with low beam divergence

Preliminary studies show that little is gained through the use of X-ray optics – beam collimation simply achieved using pin-hole

Trials are concentrating on X-ray tube rather than radioactive source (eg ^{55}Fe) since higher flux can be achieved

Initial tests using Kevex tube give 20000 X-rays s^{-1} through an 80 μm pin-hole in tungsten foil placed at 10 cm from focal spot.

Beam divergence ~0.07°



X-ray source

Further tests are being conducted using Oxford Instruments Eclipse III portable X-ray tube

Aim to trial planned micro-focus version with 50 μm spot-size later in technology programme

CCD Detectors

An X-ray diffractometer with no moving parts requires a position sensitive detector

Energy discrimination is required to identify $K\alpha/K\beta$ diffracted photons

Deep-depletion CCDs can meet this role but must have

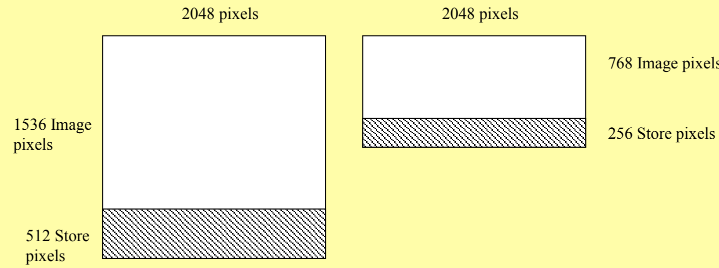
- stable cooled operating temperature (173K < T < 243K)
- low-noise operation (N <~ 25 – 30 electrons rms enc)

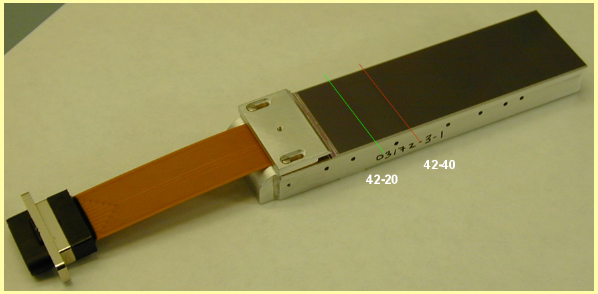
With lower noise operation (N~<10 electrons rms enc) CCD detectors can perform simultaneous X-ray diffraction and X-ray fluorescence, i.e. both the mineralogy and the elemental composition can be determined.

CCD Detectors

Custom CCD development programme underway at e2v – first devices expected end 2006

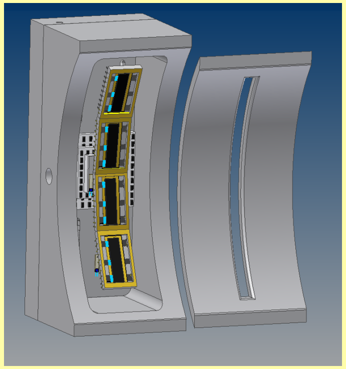
Two formats - both frame-transfer for zero dead-time – compressed frame-store to maximise collecting area

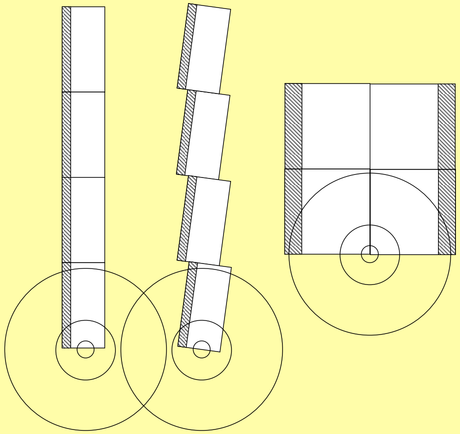




Custom package makes devices 3-sides buttable

Different array formats to be studied -Trade-off between angular resolution and collecting area



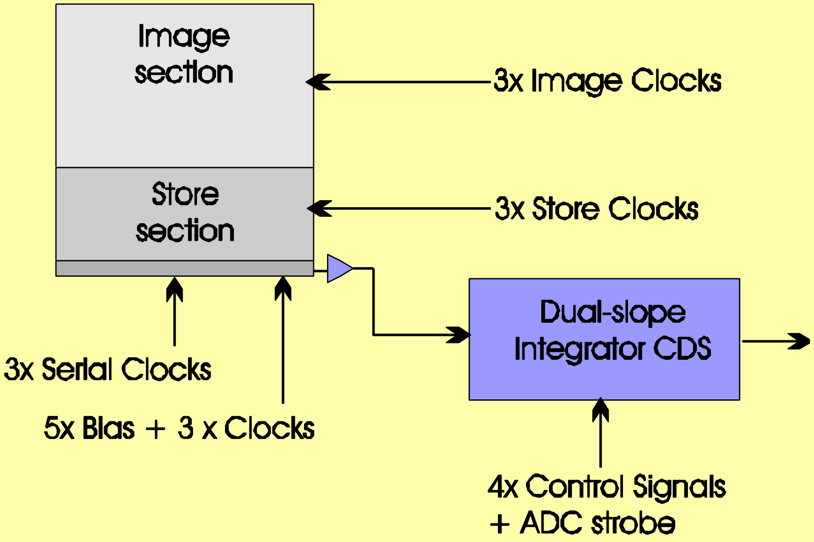


CCD Drive Electronics

CCD - Ideal detector for combined XRD-XRF, but not simple to operate

- require bias voltages, clock signals

- CCD output requires correlated double sampling (CDS) for low-noise operation



Proposed system will use FPGA for CCD clocks

– space-qualified devices readily available

- clock sequence typically fixed with few variable parameters (eg exposure time)

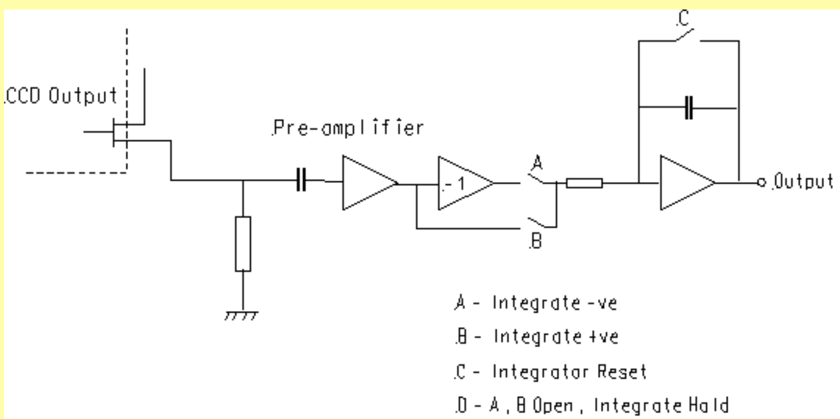
CCD Signal Processing Electronics

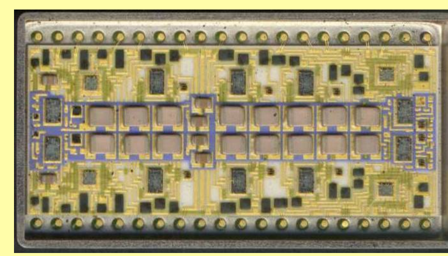
Correlated Double Sampling – various methods

Read-out speed requirement is of order 100 – 200 kHz – using dual slope integration

Breadboard trial version achieves 5 electrons rms read-noise at 100 kHz

Hybrid design complete – small size, cheaper than ASIC, built around qualified components – first trials due in Autumn





Hybrid CDS

Data Processing

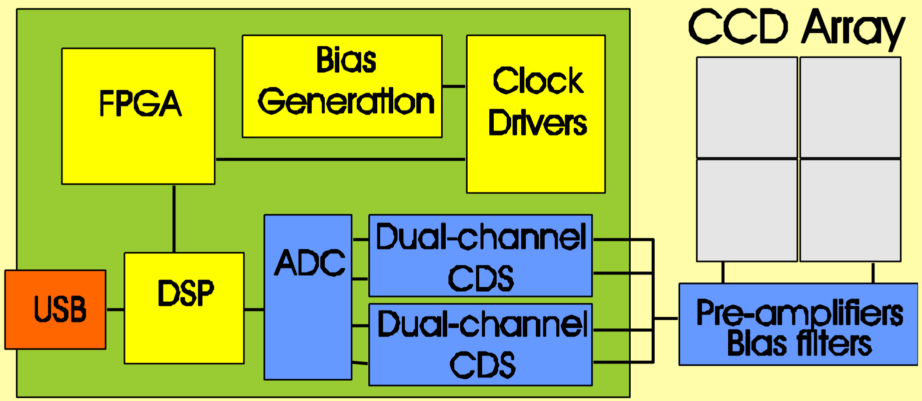
Raw data volumes from multiple CCD detectors is high

This can be reduced using real-time event processing – digital signal processor (DSP):

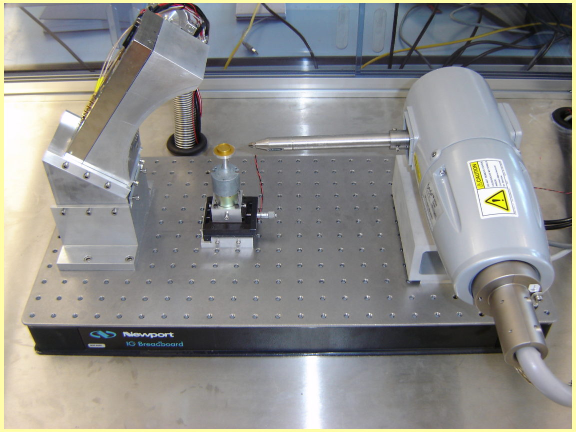
- Dynamic calibration – this is possible using the diffracted photons of known energy.
- Event reconstruction. X-ray photons that deposit their charge into more than one pixel can be 'reconstructed' to recover the original energy. These can then contribute to XRF and XRD histograms.
- Diffracted/fluoresced photon discrimination based on photon energy.
- Construction of XRF energy histogram.
- Diffraction pattern curvature correction.
- Construction of $K\alpha$ and $K\beta$ 2θ XRD diffractograms.

Prototype XRD-XRF Instrument Design

Putting it all together



Detection system

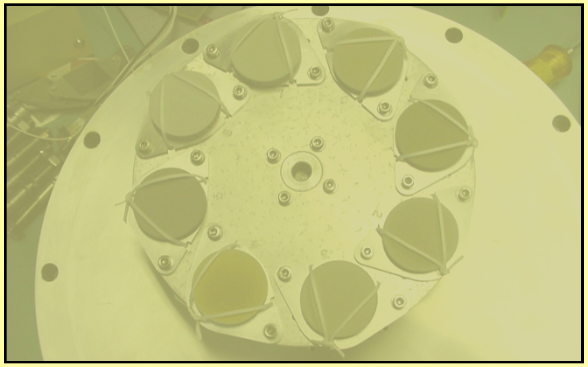


Lab XRD arrangement

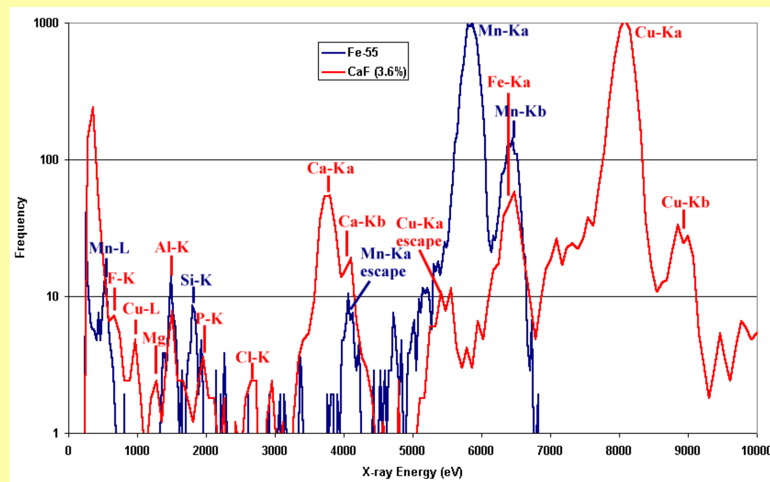
Design underway – breadboard trials started – full prototype lab testing due to begin end 2006

XRF Quantitative Analysis

Parallel programme to look at XRF quantitative analysis



Based on geological standards



Follow-on from Beagle2 XRS calibration.
Investigating angular dependence, detector QE

Summary

Activities under current funded technology development programme

(May 2006 – April 2007)

- Investigate X-ray tube technology
- Produce flight-type detector array
- Produce prototype CCD electronics
- Develop real-time XRD-XRF event detection and processing software
- Investigate detector and XRF quantitative calibration